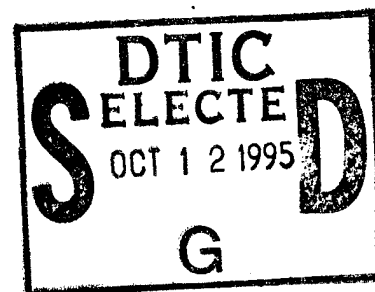


ARMY RESEARCH LABORATORY



# Fourier Transform Raman (FTR) Spectroscopy of Some Energetic Materials and Propellant Formulations II

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## 1. INTRODUCTION

Fourier transform Raman (FTR) spectroscopy using near infrared (NIR) laser light as the scattering source has been shown to be useful in the identification of crystalline components of propellant formulations (Akhaven 1991). The ease with which many principal components of propellant formulations may be identified is due to the generally good scattering characteristics of crystalline energetic materials combined with the poor scattering characteristics of most plasticizers and binders. Although using NIR laser light as the scattering source limits the signal intensity compared to that obtained using visible laser light scattering, the lack of fluorescence interference when working in the NIR spectral region often outweighs this disadvantage. If a disadvantage exists in trying to measure Raman spectra of propellant formulations using NIR laser scattering, it is that most colored formulations will absorb the laser light and combust. Fortunately, most crystalline neat energetic materials are not colored, so the technique can often yield excellent results.

Recently we published a short paper consisting of Raman spectra of several neat energetic materials and several propellant formulations (McNesby et al. 1994). It was our goal at the time to have that paper be the first in a series to aid in the assembly of a Raman spectral database of energetic materials and propellant formulations. Since that time, modifications to our instrument have enabled coverage of a broader spectral range, so we have remeasured the spectra of most of the neat energetic materials reported in the first paper, added several more spectra of neat energetic materials not measured, and also have included some new energetic formulations and techniques. We hope most readers find this report a useful continuation of our initial effort towards the characterization of energetic materials by FTR spectroscopy.

## 2. EXPERIMENTAL

The experimental apparatus has been described previously (McNesby et al. 1994). Briefly, the equipment employed in these experiments consists of a Bomem DA-8.02 Fourier transform spectrometer to which a Raman accessory has been added. Incident radiation is provided by a Quantronix Series 100 Nd:YAG laser. A simple sketch of the experimental apparatus is shown in Figure 1. Raman-shifted radiation is collected using a back-scattering geometry and is detected after filtering and interferometer modulation using a liquid nitrogen-cooled InGaAs detector. Recently the interference filters used to exclude the Rayleigh line at a wavelength of  $1.06 \mu\text{m}$  ( $9,394.5 \text{ cm}^{-1}$ ) were replaced by holographic notch

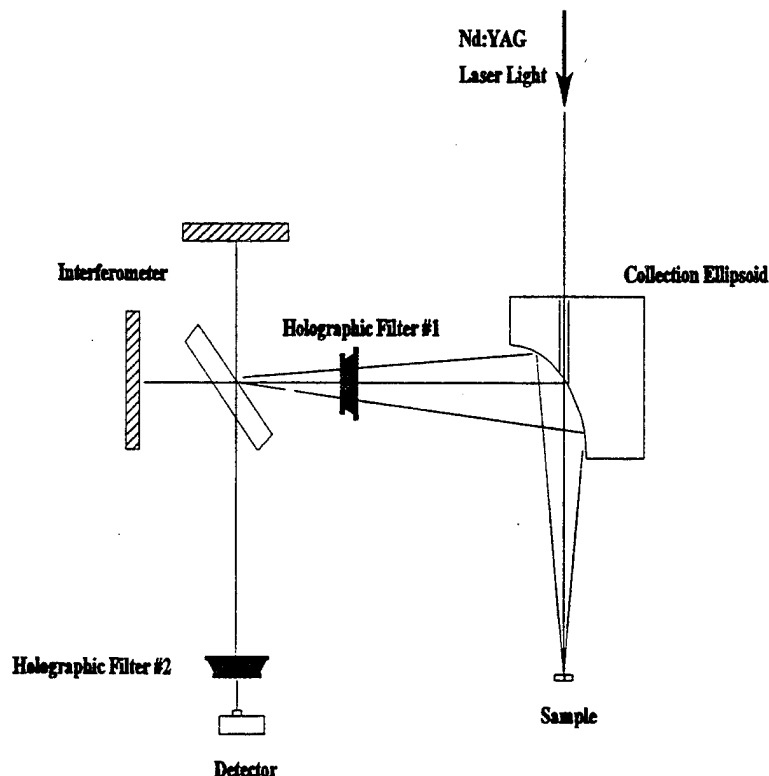


Figure 1. Simple diagram of the experimental apparatus used in these experiments.

filters (Kaiser Optical). This enabled measurement of Raman transitions shifted from 100 to 3,000  $\text{cm}^{-1}$  with respect to the Rayleigh line (the previous spectral range of the instrument was from 400 to 3,000  $\text{cm}^{-1}$ ). Also, we are able to observe anti-Stokes lines shifted from the Rayleigh line by 150 to 600  $\text{cm}^{-1}$ .

All spectra reported here were measured at 4  $\text{cm}^{-1}$  resolution using coaddition of 256 scans. Incident laser power was 400 mW. Scan time to collect each spectrum was 11 min. All spectra were measured using the back scattering arrangement shown in Figure 1. Neat samples of energetic materials were placed in 1-mm i.d. glass capillary tubes, and the Nd:YAG laser focused on the surface of the tube. Propellant formulations, when in the form of cylindrical grains, were placed in a temperature-variable holder whose position could be maintained from run to run. The Nd:YAG laser light was then focused on a flat surface of the grain created by cleaving the grain normal to its longitudinal axis. Powdered propellant formulations were placed in a 5-mm-diameter glass tube that was then placed in a sample spinner (Princeton Photonics) operated at 2,400 rpm to minimize heating effects. The Nd:YAG laser was then

focused on the surface of the spinning tube. No correction was made to any of the spectra to account for responsivity of the detector, interferometer, or filters used in the experiments. For the spectra of neat energetic materials reported here, the intense feature at zero Raman shift is due to Rayleigh scatter that was not fully attenuated by the holographic filters. The intense ringing about zero shift is a manifestation of the intensity of the Rayleigh scatter entering the interferometer and the boxcar apodization used in the Fourier transformation of the raw data (Griffith and de Haseth 1986). Samples of HNS and of the Chinese propellant formulation were provided by Atlas Wireline Services, Inc. All other propellant samples used in these experiments were obtained from in-house sources.

### 3. RESULTS AND DISCUSSION

3.1 Neat Energetic Materials. Table 1 shows a summary of the energetic materials whose FTR spectra are described in this report. Figure 2 shows the FTR spectra of the most common nitramine energetic material, cyclotrimethylenetrinitramine (RDX), and of the eight-membered ring homolog, cyclotetramethylenetetranitramine (HMX). There are several interesting features in these spectra. First, unlike our previously published spectra, we are now able to observe in each spectrum vibrational transitions to within  $100\text{ cm}^{-1}$  of the Rayleigh line. Vibrational transitions due to ring deformations in RDX and HMX, occurring at Raman shifts of from  $100$  to  $400\text{ cm}^{-1}$ , were not measurable in our laboratory prior to installation of the holographic rejection filters. Crystal lattice modes (Goetz and Brill 1979) in nitramine crystals, which occur below  $100\text{ cm}^{-1}$ , fall within the "notch" of the holographic filters so are not observable. In the FTR spectrum of HMX, an intense peak is observed that is centered at a Raman shift of  $150\text{ cm}^{-1}$ . This peak is close to the intensity of the most intense ring stretch features that occur in HMX from  $800$  to  $1,000\text{ cm}^{-1}$ .

Figure 3 shows the spectrum of the nitrate ester pentaerythritol tetranitrate (PETN), a common ingredient in some commercial explosives. Also shown is the FTR spectrum of nitroguanidine (NQ). The N-H stretch in nitroguanidine at a Raman shift of approximately  $3,200\text{ cm}^{-1}$  is not observed since it occurs beyond the range of the InGaAs detector. Figure 4 shows the spectrum of ammonium dinitramide (ADN), an oxidizer whose use is rapidly expanding within the propellant community, and of trinitrotoluene (TNT). In both of these spectra, features below  $400\text{ cm}^{-1}$  are among the most intense in each spectrum. For the spectrum of TNT, the slightly elevated baseline is caused by sample heating during measurement of the spectrum since neat TNT is light yellow in color and absorbs some of the incident Nd:YAG laser radiation.

Table 1. Energetic Materials Used in These Experiments

Energetic Material	Laser Power (mW)	Composition
RDX	400	neat
HMX	400	neat
PETN	400	neat
NQ	400	neat
ADN	400	neat
TNT	400	neat
CL-20	400	neat
TNAZ	400	neat
HNS	400	neat
pDNPA	400	neat
NC	400	neat
DMNA	400	neat
AP	400	neat
GAP	400	neat
XM39	400	76% RDX, 24% binders and plasticizers

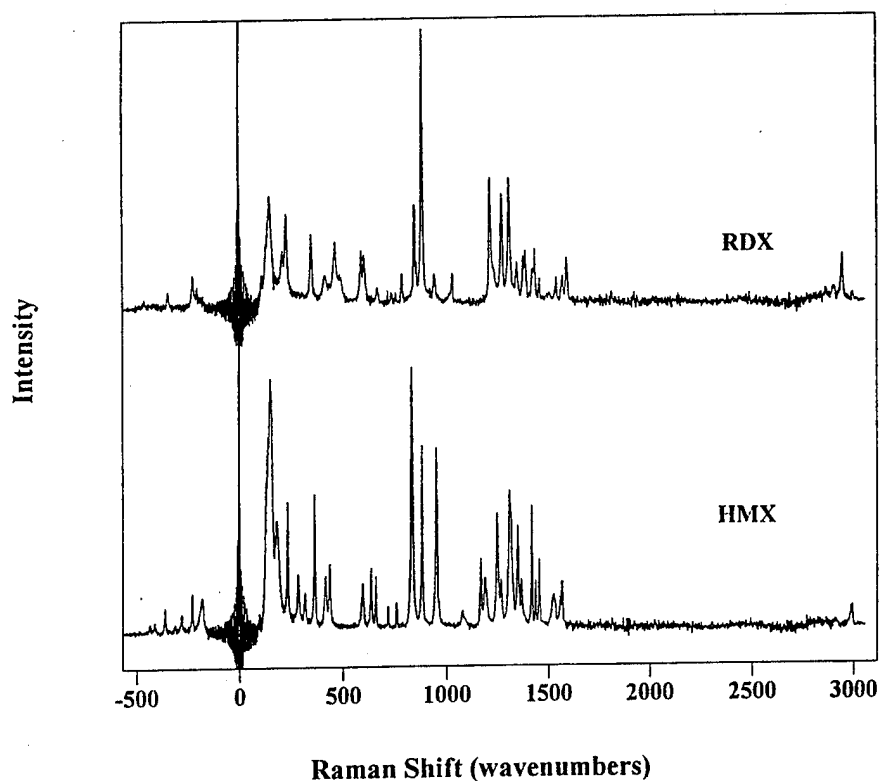


Figure 2. The Raman spectra of RDX and HMX.

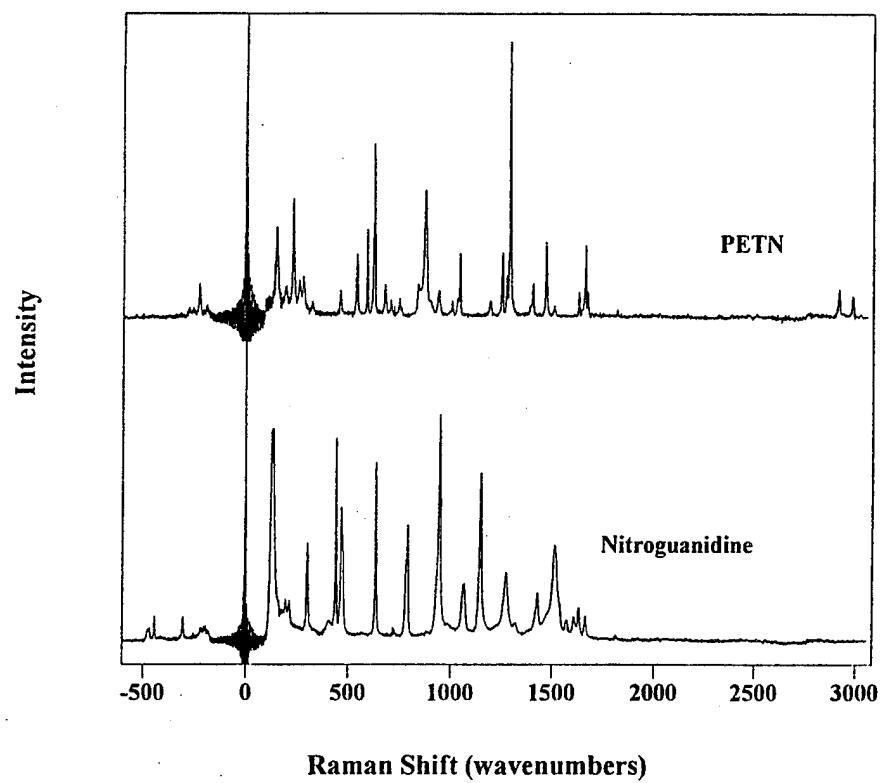


Figure 3. The FTR spectra of PETN and NQ.

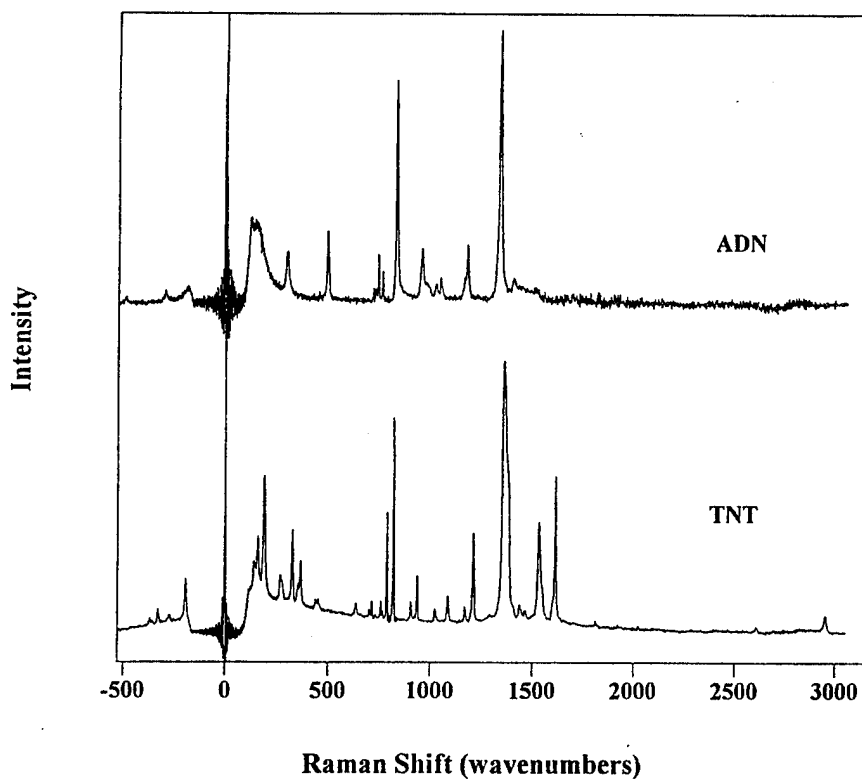


Figure 4. The FTR spectra of ADN and TNT.

Figure 5 shows the spectra of beta-hexanitrohexaazaisowurtzitane (CL-20, previously reported using the alternate name b-HNIW), an energetic caged nitramine, and 1, 3, 3-trinitroazetidine (TNAZ), a recently developed energetic nitramine containing a germinal dinitro group. Figure 6 shows the spectrum of hexanitrostilbene (HNS), an industrial explosive used in high-temperature applications, and polydinitropropyladapate (pDNPA), an energetic polymeric binder used in some propellant formulations. Both spectra were measured using incident radiation at a power of 400 mW, although the signal-to-noise ratio in the spectrum of HNS is superior to that of the spectrum of pDNPA. These spectra illustrate the general observation that crystalline energetic materials are better Raman scatterers than polymeric energetic materials (McNesby et al. 1994).

Figure 7 shows the FTR spectra of dimethylnitramine (DMNA), a simple energetic nitramine, and nitrocellulose (NC). While both spectra were measured using the same incident laser power and using the same sampling technique (1-mm i.d. capillary tube), the signal-to-noise ratio in the spectrum of crystalline DMNA is superior to that of polymeric nitrocellulose. Both samples were in the form of white powders. Figure 8 shows the FTR spectra of glycidyl azide polymer (GAP), an energetic binder, and ammonium perchlorate (AP), an important oxidizer for many composite propellant formulations, and the only inorganic energetic material included in this study. Again, it may be seen from Figure 8 that the FTR spectrum of the crystalline material (AP) has a higher signal-to-noise ratio than the polymeric GAP.

**3.2 Applications to Propellant Analysis.** Figure 9 shows the FTR spectra of RDX, HMX, and XM39, a propellant formulation whose principal ingredient is RDX. The spectrum of XM39 is an illustration of the utility of FTR spectroscopy for energetic material characterization in general and for identification of nitramine-based explosives in particular. While the spectra of RDX and HMX in Figure 2 were measured using neat powdered samples in capillary tubes, the spectrum of XM39 was measured by scattering the Nd:YAG laser light off of the interior surface of a cleaved propellant grain. Even with this simple sampling technique, assignment of RDX as the principal crystalline energetic ingredient in XM39 is unambiguous. Shown in the spectrum of XM39 is evidence of HMX "impurity" in the formulation. HMX is a normal by-product of RDX synthesis in most domestically produced RDX. Also shown in the spectrum of XM39 is the increased intensity (relative to neat RDX) of the feature near  $150\text{ cm}^{-1}$ . This increased intensity (see Figure 2) may be due to the small HMX "impurity" in domestically manufactured RDX.



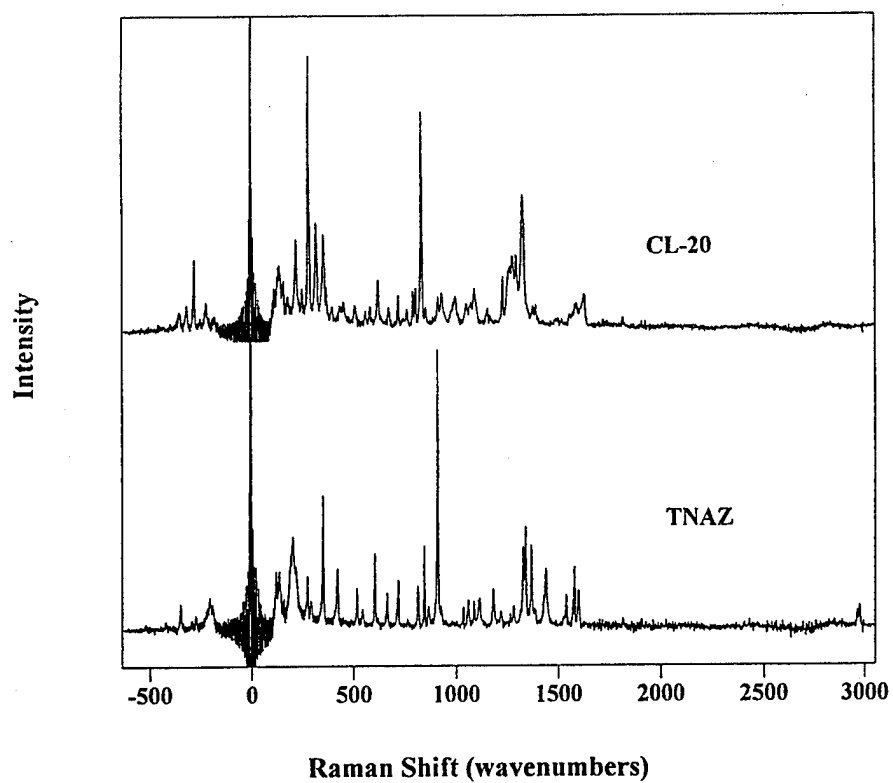


Figure 5. The FTR spectra of CL-20 and TNAZ.

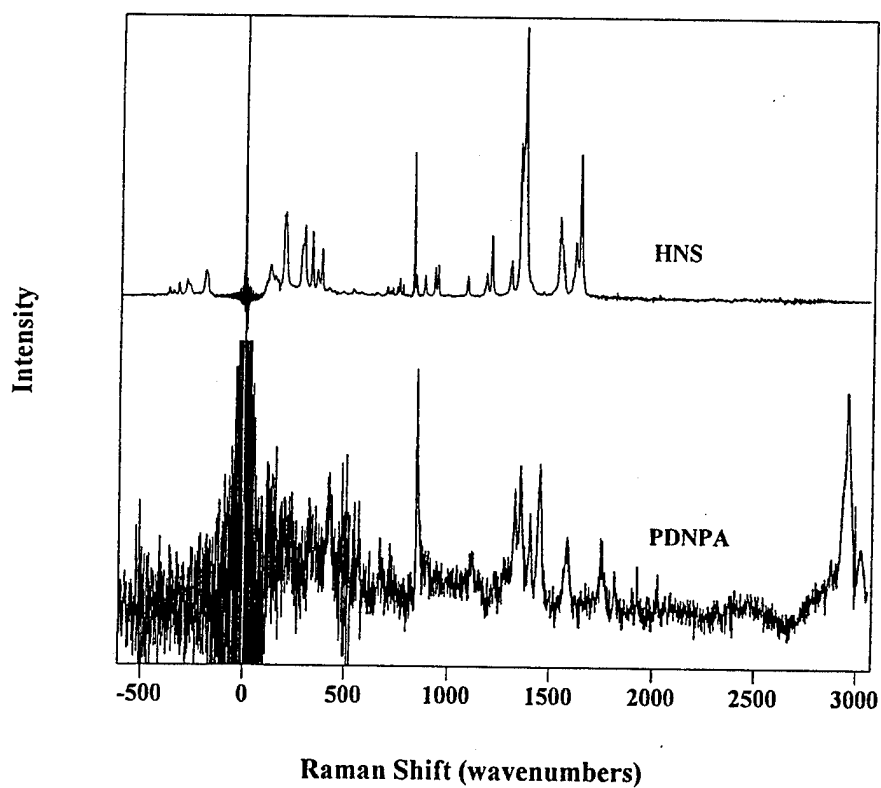


Figure 6. The FTR spectra of HNS and pDNPA.

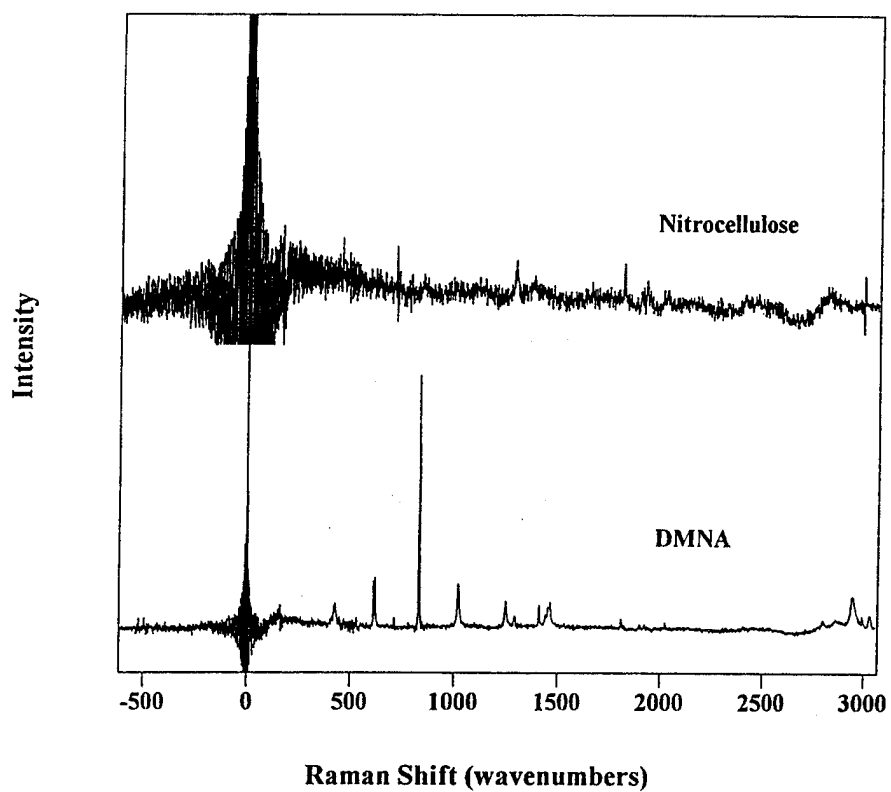


Figure 7. The FTR spectra of DMNA and NC.

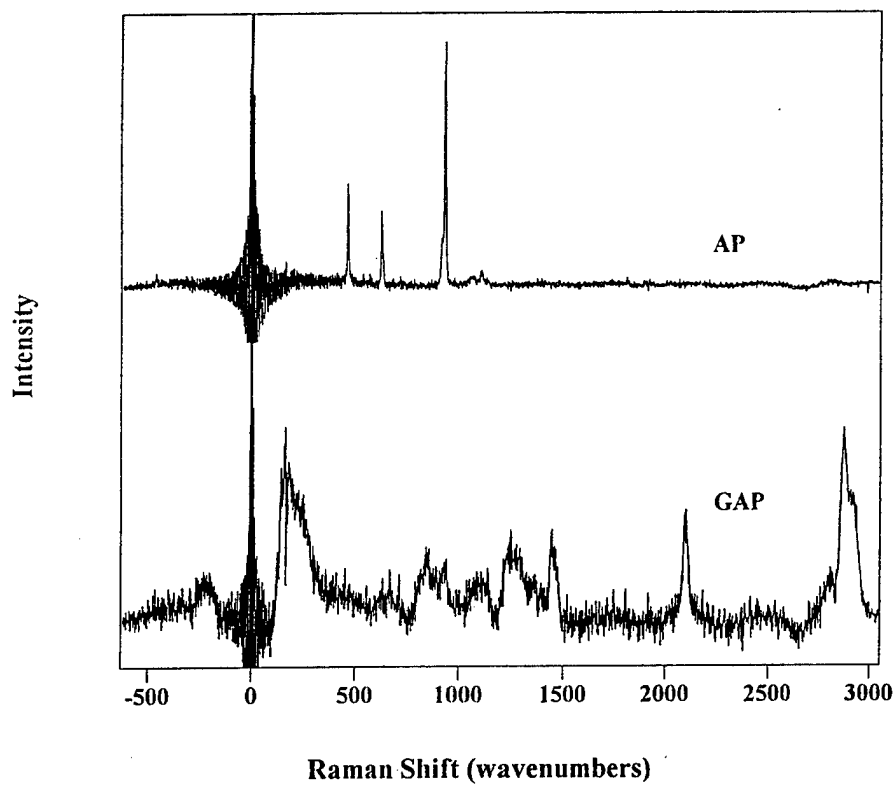


Figure 8. The FTR spectra of GAP and AP.

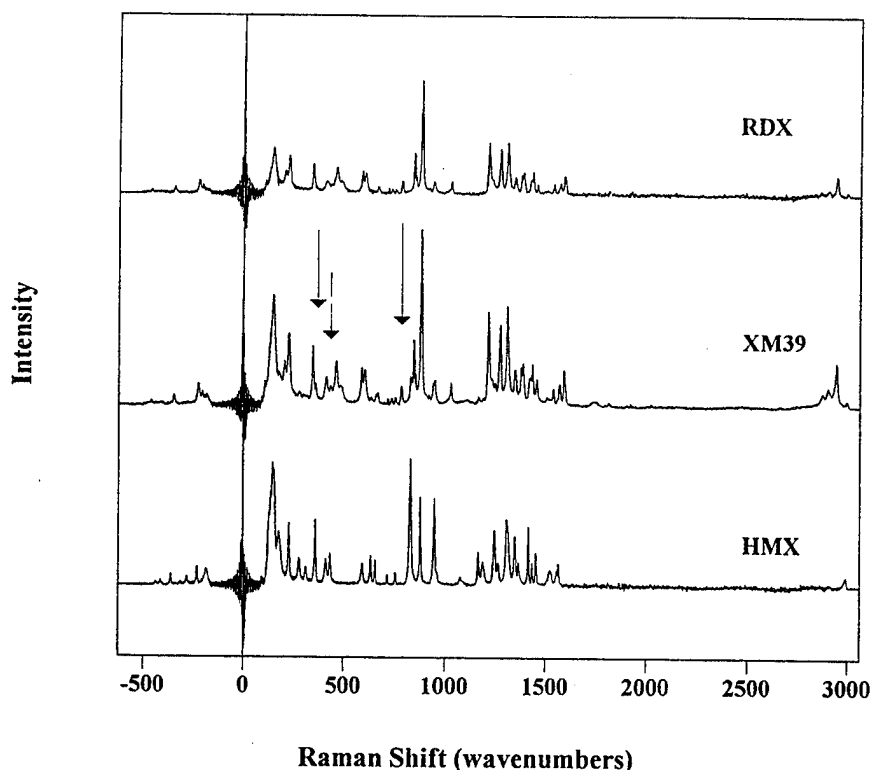


Figure 9. The Raman spectra of RDX, HMX, and XM39. Arrows indicate features in the spectrum of XM39 due to slight HMX "impurities" in domestically manufactured RDX.

Figure 10 shows the Raman spectra of RDX, XM39, and a commercially available propellant formulation believed to be manufactured in mainland China. Since the Chinese propellant formulation had a thin graphite coating that would cause the sample to absorb some of the incident laser radiation, the spectrum was measured by scattering the incident Nd:YAG laser radiation off of the surface of a spinning (2,400 rpm) 5-mm i.d. glass tube containing the propellant formulation. From Figure 10, it is apparent that the principal crystalline energetic material in the Chinese propellant formulation is RDX. Also, the spectrum of the Chinese propellant formulation shows no evidence of HMX, suggesting that the RDX present in the Chinese propellant formulation was manufactured by a different process than domestically produced RDX.

Figure 11 shows a portion of the Raman spectrum of the propellant formulation XM39 as it is heated from room temperature to the onset of decomposition at 458 K (185° C). This spectrum was measured by scattering the Nd:YAG laser light off the surface of a slice of XM39 taken from the interior of a propellant grain. As shown previously, the principal crystalline energetic ingredient in XM39 is RDX.

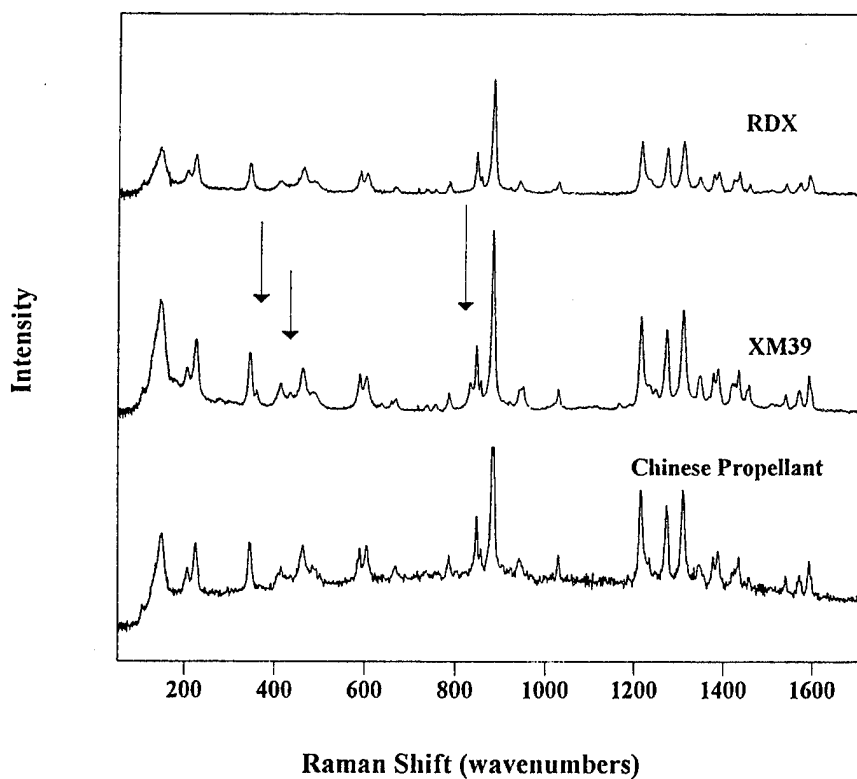


Figure 10. The FTR spectra of RDX, XM39, and a propellant formulation believed to have been manufactured in mainland China. Arrows indicate the presence of HMX in XM39.

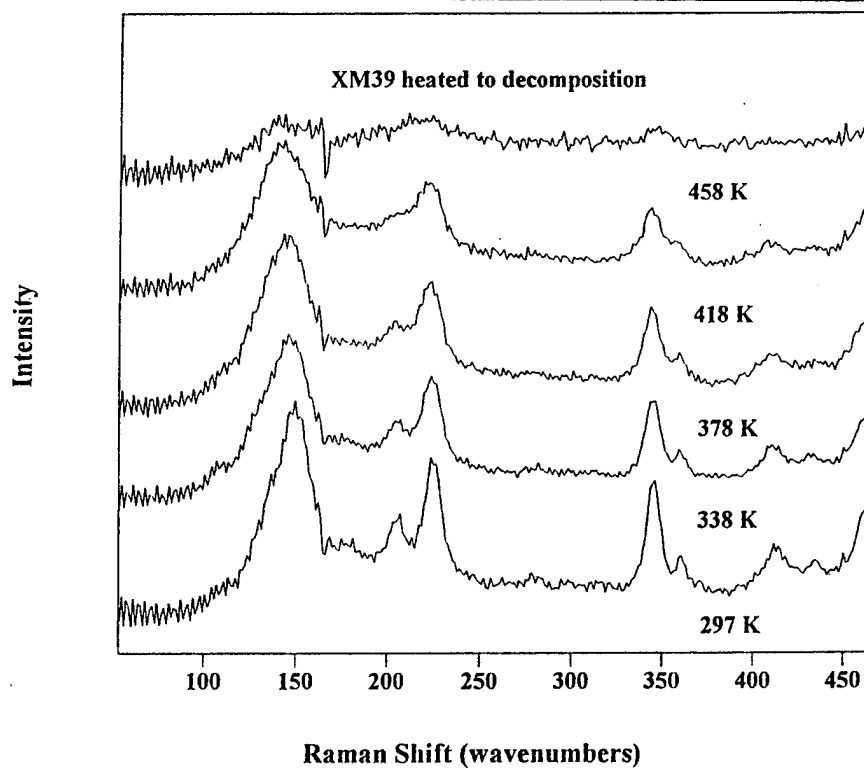


Figure 11. The FTR spectra of XM39 as it is heated from 297 K to 458 K. Note the slight red shift of the feature near 150  $\text{cm}^{-1}$ .

For XM39, as temperature is increased, the scattering intensity decreases (as shown by the decreased signal to noise ratio in the spectra) and there is a slight red shift of the feature near  $150\text{ cm}^{-1}$ . This red shift was most notable for features at low Raman frequencies, and was negligible at Raman frequencies above  $500\text{ cm}^{-1}$ . As the sample temperature approaches the melting point of RDX (477 K), the Raman intensity rapidly diminishes, reflecting a loss of crystallinity in the principal energetic ingredient in the formulation.

#### 4. CONCLUSION

It has been shown that FTR spectroscopy using NIR laser radiation as the scattering source can be a useful tool in characterizing many neat samples of energetic materials and of some propellant formulations. Raman spectra of some common energetic materials, as well as some newly formulated energetic materials, have been measured from  $100$  to  $3,000\text{ cm}^{-1}$ . To our knowledge, Raman spectra measured to within  $100\text{ cm}^{-1}$  of the Rayleigh line have not been reported for most energetic materials. Perhaps the most interesting result from our ability to measure Raman spectra over a wider spectral range is the observation that the most intense peak in the HMX spectrum occurs near  $150\text{ cm}^{-1}$ . Finally, we believe that for many samples of energetic materials containing crystalline principal ingredients, FTR spectroscopy using NIR laser scattering should be the preferred choice of investigators for forensic analysis when adequate sample is present. We are presently extending the technique to the investigation of energetic materials at elevated temperatures, in formulations, and in solutions.

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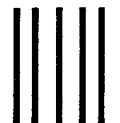
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